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## IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of  
RATH et al.

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) Art Unit:1713

Serial No. 09/701,587

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) Examiner: Lu

Filed: November 30, 2000

For: PREPARATION OF HIGHLY REACTIVE POLYISOBUTENES

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DECLARATION UNDER 37 CFR §1.132

I, Hans-Peter Rath, Dr., a citizen of the Federal Republic of Germany and residing at Friedhofstrasse 7, 67269 Grünstadt, Germany, declare as follows:

I am a fully trained chemist, having studied chemistry at Technische Universität Aachen (Technical University of Aachen), Germany, during the period of 1965-1974.

I obtained my doctor's degree from Technische Universität Aachen in 1974.

Since 1975, when I joined BASF Aktiengesellschaft of 67056 Ludwigshafen, Germany, I have been working primarily in the field of cationic polymerization.

Therefore, I am familiar with the field to which the subject application relates. I am also familiar with the examiner's rejections of the claims of the subject application.

In the present Declaration, I state the results of Reactivity Tests comparing that of the instant invention with that of a comparative Example.

As outlined in the table below, the following process according to the instant invention and according to US 5,408,018, were compared. Example A as outlined in the table below corresponds to Example 7 of the instant application on page 13, lines 42-45 and in the table on page 14. The example A, corresponding to example 7 of the

application was carried out as follows.

Applicants utilized a recycle reactor consisting of a Teflon tube which had a length of 7.1 m and an internal diameter of 6 mm via which 100 l/h of the reactor content were circulated by means of a gear pump. The tube and pump had a capacity of 200 ml. The Teflon tube and the pump head were immersed in a cooling bath having a temperature of  $-23.8^{\circ}\text{C}$  (cryostat). A mixture of 300 g/h of isobutene and 300 g/h of hexane was dried over a  $3\text{\AA}$  molecule sieve to a water content of less than 3 ppm, and then fed to the recycle reactor through a capillary which had an internal diameter of 2 mm and which was pre-cooled to  $-23^{\circ}\text{C}$ .  $\text{BF}_3$  and isopropanyl/diisopropylether as complexing agents were directly introduced into the hexane feed to the reactor. The feed of 10 mmol/h isopropanol and 25 ml/h di-sec-butyl ether and n-hexane (as diluent) was set to 15:1:4, and the amount of  $\text{BF}_3$  was varied until a concentration of isobutene of 1% by weight was obtained.

At a reactor temperature of  $-18^{\circ}\text{C}$  the  $\text{BF}_3$  feed amounted to 21.4 mmol/h. The reactor effluent was washed with water and worked up by distillation at  $230^{\circ}\text{C}/2\text{mbar}$ . The molecular weight  $M_n$  of the polymer was 890, the reactivity (vinylidene double bond content) was 95.9%.

This process was repeated in the comparative example utilizing isopropyl tert-butyl ether, as disclosed in US 5,408,018, was used instead of di-sec-butyl ether. To arrive at a molecular weight in the magnitude obtained in Example A, the amounts of  $\text{BF}_3$  and isopropanol had to be increased over Example A. Thus, as you can see from the results displayed in table form below, the instant process provides an unexpected increase in reactivity over that of the process utilizing isopropyl tert-butyl ether.

The following example shows the results of these tests:

Example	BF <sub>3</sub> [mmol/h]	Isopropanol [mmol/h]	Ether [mmol/h]	Molar Ratios		[Isobutene] weight %	M <sub>n</sub>	Reactivity [%]
				B:I	(I+B):S			
A	21.4	10.0	25.0 Di-sec- butylether	2.5	1.64	1.0	890	95.9
compara tive example	29.7	15.0	25.0 Isopropyl- tert- butylether	2.5	1.36	1.0	960	85.4

Signed at 67056 Ludwigshafen, Germany, this 1. day of Jan, 2003



Hans-Peter Rath

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